

FORM PTO-1390 (REV 11-2000)	U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE	ATTORNEY'S DOCKET NUMBER 30-551
TRANSMITTAL LETTER TO THE UNITED STATES DESIGNATED/ELECTED OFFICE (DO/EO/US) CONCERNING A FILING UNDER 35 U.S.C. 371		U.S. APPLICATION NO. (If known, see 37 C.F.R. 1.5) 09/889486 Unknown
INTERNATIONAL APPLICATION NO. PCT/FI00/00045	INTERNATIONAL FILING DATE 20 January 2000	PRIORITY DATE CLAIMED 22 January 1999
TITLE OF INVENTION TREATMENT OF RECIRCULATED FILTRATES IN PULP PRODUCTION		
APPLICANT(S) FOR DO/EO/US PIKKA et al		
Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:		
1. <input checked="" type="checkbox"/> This is a FIRST submission of items concerning a filing under 35 U.S.C. 371. 2. <input type="checkbox"/> This is a SECOND or SUBSEQUENT submission of items concerning a filing under 35 U.S.C. 371. 3. <input checked="" type="checkbox"/> This is an express request to begin national examination procedures (35 U.S.C. 371(f)). The submission must include items (5), (6), (9) and (21) indicated below. 4. <input checked="" type="checkbox"/> The U.S. has been elected by the expiration of 19 months from the priority date (Article 31). 5. A copy of the International Application as filed (35 U.S.C. 371(c)(2)). a. <input type="checkbox"/> is attached hereto (required only if not communicated by the International Bureau). b. <input checked="" type="checkbox"/> has been communicated by the International Bureau. c. <input type="checkbox"/> is not required, as the application was filed in the United States Receiving Office (RO/US). <input type="checkbox"/> An English language translation of the International Application as filed (35 U.S.C. 371(c)(2)). a. <input type="checkbox"/> is attached hereto. b. <input type="checkbox"/> has been previously submitted under 35 U.S.C. 154(d)(4). <input checked="" type="checkbox"/> Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3)). a. <input type="checkbox"/> are attached hereto (required only if not communicated by the International Bureau). b. <input checked="" type="checkbox"/> have been communicated by the International Bureau. c. <input type="checkbox"/> have not been made; however, the time limit for making such amendments has NOT expired. d. <input type="checkbox"/> have not been made and will not be made. <input type="checkbox"/> An English language translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)). 9. <input type="checkbox"/> An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)). 10. <input type="checkbox"/> A English language translation of the annexes of the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)).		
Items 11 To 20 below concern document(s) or information included:		
11. <input checked="" type="checkbox"/> An Information Disclosure Statement under 37 C.F.R. 1.97 and 1.98. 12. <input type="checkbox"/> An assignment document for recording. A separate cover sheet in compliance with 37 C.F.R. 3.28 and 3.31 is included. 13. <input checked="" type="checkbox"/> A FIRST preliminary amendment. 14. <input type="checkbox"/> A SECOND or SUBSEQUENT preliminary amendment. 15. <input type="checkbox"/> A substitute specification. 16. <input type="checkbox"/> A change of power of attorney and/or address letter. 17. <input type="checkbox"/> A computer-readable form of the sequence listing in accordance with PCT Rule 13ter.2 and 35 U.S.C. 1.821-1.825. 18. <input type="checkbox"/> A second copy of the published international application under 35 U.S.C. 154(d)(4). 19. <input type="checkbox"/> A second copy of the English language translation of the international application under 35 U.S.C. 154(d)(4). 20. <input type="checkbox"/> Other items or information.		

U.S. APPLICATION NO. (If known, use 37 C.F.R. 1.5) 097889486 Unknown	INTERNATIONAL APPLICATION NO. PCT/FI00/00045	ATTORNEY'S DOCKET NUMBER 30-551
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21. ☒ The following fees are submitted:

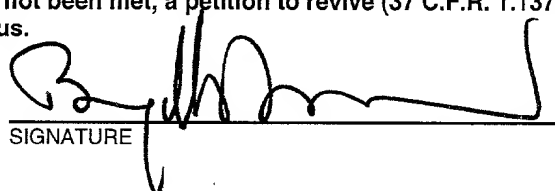
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-- Neither international preliminary examination fee (37 C.F.R. 1.482) nor international search fee (37 C.F.R. 1.445(a)(2)) paid to USPTO and International Search Report not prepared by the EPO or JPO\$1000.00 -- International preliminary examination fee (37 C.F.R. 1.482) not paid to USPTO but International Search Report prepared by the EPO or JPO.....\$860.00 -- International preliminary examination fee (37 C.F.R. 1.482) not paid to USPTO but international search fee (37 C.F.R. 1.445(a)(2)) paid to USPTO\$710.00 -- International preliminary examination fee (37 C.F.R. 1.482) paid to USPTO but all claims did not satisfy provisions of PCT Article 33(1)-(4).....\$690.00 -- International preliminary examination fee (37 C.F.R. 1.482) paid to USPTO and all claims satisfied provisions of PCT Article 33(1)-(4).....\$100.00																																					
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Surcharge of \$130.00 for furnishing the oath or declaration later than <input type="checkbox"/> 20 <input checked="" type="checkbox"/> 30 months from the earliest claimed priority date (37 C.F.R. 1.492(e)).	\$	130.00																																			
<table border="1" style="width:100%; border-collapse: collapse;"> <thead> <tr> <th style="width:20%;">CLAIMS</th> <th style="width:10%;">NUMBER FILED</th> <th style="width:10%;">NUMBER EXTRA</th> <th style="width:10%;">RATE</th> <th style="width:10%;"></th> <th style="width:10%;"></th> <th style="width:10%;"></th> </tr> </thead> <tbody> <tr> <td>Total Claims</td> <td>17</td> <td>-20 =</td> <td>0</td> <td>X</td> <td>\$18.00</td> <td>\$ 0.00</td> </tr> <tr> <td>Independent Claims</td> <td>1</td> <td>-3 =</td> <td>0</td> <td>X</td> <td>\$80.00</td> <td>\$ 0.00</td> </tr> <tr> <td colspan="5">MULTIPLE DEPENDENT CLAIMS(S) (if applicable)</td> <td>\$270.00</td> <td>\$ 0.00</td> </tr> <tr> <td colspan="5" style="text-align: right;">TOTAL OF ABOVE CALCULATIONS =</td> <td>\$</td> <td>1130.00</td> </tr> </tbody> </table>	CLAIMS	NUMBER FILED	NUMBER EXTRA	RATE				Total Claims	17	-20 =	0	X	\$18.00	\$ 0.00	Independent Claims	1	-3 =	0	X	\$80.00	\$ 0.00	MULTIPLE DEPENDENT CLAIMS(S) (if applicable)					\$270.00	\$ 0.00	TOTAL OF ABOVE CALCULATIONS =					\$	1130.00		
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<input checked="" type="checkbox"/> Applicant claims small entity status. See 37 CFR 1.27. The fees indicated above are reduced by 1/2.		0.00																																			
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Processing fee of \$130.00, for furnishing the English Translation later than <input type="checkbox"/> 20 <input type="checkbox"/> 30 months from the earliest claimed priority date (37 C.F.R. 1.492(f)).		0.00																																			
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Fee for recording the enclosed assignment (37 C.F.R. 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 C.F.R. 3.28, 3.31). \$40.00 per property	+	\$ 0.00																																			
Fee for Petition to Revive Unintentionally Abandoned Application (\$1240.00 - Small Entity = \$620.00)		\$ 0.00																																			
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a. ☒ A check in the amount of \$1130.00 to cover the above fees is enclosed.
 b. ☐ Please charge my Deposit Account No. 14-1140 in the amount of \$_____ to cover the above fees. A duplicate copy of this form is enclosed.
 c. ☒ The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. 14-1140. A duplicate copy of this form is enclosed.
 d. ☒ The entire content of the foreign application(s), referred to in this application is/are hereby incorporated by reference in this application.

NOTE: Where an appropriate time limit under 37 C.F.R. 1.494 or 1.495 has not been met, a petition to revive (37 C.F.R. 1.137(a) or (b)) must be filed and granted to restore the application to pending status.

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 SIGNATURE

Bryan H. Davidson
 NAME

30,251
 REGISTRATION NUMBER

July 18, 2001
 Date

09/889486

JC18 Rec'd PCT/PTO 1 8 JUL 2001

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Patent Application of

PIKKA et al

Atty. Ref.: **30-551**

Serial No. **Unknown**

Group:

National Phase of: **PCT/FI00/00045**

International Filing Date: **20 January 2000**

Filed: **July 18, 2001**

Examiner:

For: **TREATMENT OF RECIRCULATED FILTRATES IN PULP
PRODUCTION**

* * * * *

July 18, 2001

Assistant Commissioner for Patents
Washington, DC 20231

Sir:

PRELIMINARY AMENDMENT

Prior to calculation of the filing fee and in order to place the above identified application in better condition for examination, please amend the claims as follows:

IN THE CLAIMS

Please substitute the following amended claims for corresponding claims previously presented. A copy of the amended claims showing current revisions is attached.

7. (Amended) A method as claimed in claim 1, characterized in that at least one of the washers (8, 12, 16) is a washer or a press from which at least either at least two filtrates (FC, FD) having different physical properties are extracted or to which at least two filtrates having different physical properties are introduced.

PIKKA et al
Serial No. **Unknown**

REMARKS

Attached hereto is a marked-up version of the changes made to the claims by the current amendment. The attached page is captioned "**Version with markings to show changes made.**"

The above amendments are made to place the claims in a more traditional format.

Respectfully submitted,

NIXON & VANDERHIE P.C.

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VERSION WITH MARKINGS TO SHOW CHANGES MADE

7. (Amended) A method as claimed in [any of the preceding claims] claim 1, characterized in that at least one of the washers (8, 12, 16) is a washer or a press from which at least either at least two filtrates (FC, FD) having different physical properties are extracted or to which at least two filtrates having different physical properties are introduced.

ART 34 AMDT

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METHOD FOR TREATMENT OF PULP

The present invention relates to a method for treatment of pulp. The invention is particularly well applicable in intensifying the washing of fiber suspensions in the paper manufacturing industry in applications, which utilize liquid recycled from later washing stages of the process as wash liquid. According to a preferred embodiment of the invention, a part of the liquid to be fed to a washer is separated to a flow of its own and divided into a cleaner and a fouler fraction which are then recycled to appropriate locations in the process. According to another preferred embodiment, condensate from evaporation or liquid cleaned otherwise is fed to a point of the process, which aims at increasing the purity of the pulp as much as possible. The liquid obtained for the purification is filtrate from the feed of an evaporation plant, i.e. chemical recovery, from the liquid circulation of the chemical recovery, from the liquid circulation of the digestion plant, from the circulation of the washing plant, from the wash circulation of an oxygen stage, or from the wash circulation of the bleach plant.

The tendency in the wood processing industry has for decades been to reduce the water consumption of the pulp bleaching process and the related washing states. This has resulted in the introduction of the so-called counter-current washing. In counter-current washing, clean wash water is introduced into the last bleaching stage of a pulp treatment line for use as the wash liquid and filtrate obtained from this washing stage is brought to the preceding washing stage for use as the wash liquid, and so on. At its best, the process enables the liquid to circulate through the whole process and end up via the digester to the chemical recovery in the evaporation plant. In other words, the most modern plants may have a wash liquid circulation into which no clean liquid need be introduced from outside the plant and no liquid is discharged from the washing circulation before the digestion process.

However, it has been discovered in the newest plants that the quality of pulp, for example in terms of pulp strength, tends to deteriorate in various treatment stages more than in mills using more liquid. This has been noticed first in connection with the so-called oxygen bleaching stage when the oxygen bleaching stage follows brown stock washing.

Therefore, it has been concluded that impurities dissolved from the pulp to the liquid phase come to the oxygen stage from the digestion plant, from the oxygen stage itself because of the recirculation of filtrates, and also from later bleaching stages. This impurity may be measured for example based on dry solids, sodium, COD or other known parameters.

These problems, i.e. primarily the decrease in the strength of the pulp and secondarily the high dry-solids content in the wash liquid and thus in the pulp, have been solved by developing a solution according to which a part of the liquid circulated counter-currently is separated to form a partial flow which is treated in order to at least partly separate dry solids from the liquid, in other words to divide the liquid into a cleaner and a fouler fraction. The fouler one of these fractions is returned to the pulp at a point where the dry solids content of the liquid phase of the fiber suspension is at least the same as that of the fouler fraction to be introduced. This point may be somewhere in the process between the digester and the recovery, in the digester itself, or after the digestion plant. Correspondingly, the cleaner fraction is returned to a point in the process where it is most beneficial. The volume of the dissolved inorganic and organic material, i.e. impurities, introduced into the oxygen stage may be reduced by extracting foul wash filtrate and introducing cleaner liquid or by introducing more cleaner liquid before the oxygen stage.

According to a preferred embodiment of the invention the greatest advantage is to be gained by introducing the cleaner fraction straight to the point from which the partial flow to be cleaned was separated.

According to another preferred embodiment of the invention the cleaner fraction is returned to a washing stage as late in the process as possible whereby its purity (compared to the rest of the wash liquid) has effect in as many washing stages as possible.

According to a third preferred embodiment of the invention the cleaner fraction is returned to the point in the process where its effect is desired to be the strongest.

According to a fourth preferred embodiment of the invention the cleaner fraction is divided so that it is distributed to several points in the process.

5 Further, according to a preferred embodiment of the invention the fouler fraction (concentrate) is returned to a point of the process in which the dry solids content (foulness) of the liquid phase is at least the same as that of the concentrate returned.

The method of the invention may employ for example one or more single-step or multi-step evaporator/s, membrane separator/s or any other separator suitable for the purpose.

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Other characteristic features of the method and the apparatus are disclosed in the appended patent claims.

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The method of the invention is described more in detail below with reference to the accompanying drawing figures of which

Fig. 1 illustrates a so-called prior art fiber line for treatment of chemical pulp;

Fig. 2 illustrates a solution according to a preferred embodiment of the invention for treatment of filtrate/wash liquid;

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Fig. 3 illustrates a solution according to a second and a third preferred embodiment of the invention for treatment of filtrate/wash liquid in connection with a continuous digester;

Fig. 4 illustrates a solution according to further six preferred embodiments of the invention for treatment of filtrate/wash liquid in connection with a continuous digester;

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Fig. 5 illustrates a solution according to further four preferred embodiments of the invention for treatment of filtrate/wash liquid in connection with a batch digester;

Fig. 6 illustrates a solution according to further six preferred embodiment of the invention for treatment of filtrate/wash liquid in connection with a batch digester;

Fig. 7 illustrates the influence of the COD content on the consumption of bleaching chemical;

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Fig. 8 illustrates the influence of the COD content on the decrease of viscosity in the oxygen stage; and

Fig. 9 illustrates the solubility of soap as a function of the dry solids content of the black liquor.

As illustrated in Fig. 1, a prior art fiber line, i.e. a line used for producing and bleaching pulp, comprises the following components. The first component on the left is a digestion plant referred to by the reference number 2 which may comprise one or more digester/s. If the process is the so-called continuous digestion as in Fig. 1 there is only one digester, and if the process is the so-called batch digestion (illustrated in Fig. 5 and 6 in connection with some preferred embodiments of the invention), there are several digesters, usually in the order of 5 - 10. The digestion plant is usually followed in both cases by a so-called blow tank 4. In a batch digestion process the blow tank is indispensable as the digesters of a batch digestion process are discharged one at a time to a blow tank, from which a continuous and even pulp flow is taken to the subsequent process. In the process, the blow tank 4 is usually followed by a screening plant 6 in which the particles not acceptable in the produced pulp are separated from the pulp. The screening plant may be located also somewhere else in the process as will be described later. The screening plant 6 is followed by so-called brown stock washing 8, which may be performed with a DRUMDISPLACER® washer (illustrated in Fig. 1), a diffuser, a pressure diffuser, one or several suction drum filter/s, one or several pressure filters, presses, other equipment available in the market intended for washing pulp or any combination of these. In some processes, the screening plant 6 may be arranged to follow the brown stock washing.

The following stage in the process illustrated in the figure is oxygen delignification 10, which today more and more often is performed in a two-vessel reactor, i.e. in two steps, as illustrated in the figure, and it is followed by an oxygen stage washing 12. After this the process continues in alternating different bleaching stages and washes separating these until the pulp is bright adequate for the purpose intended.

The process works so that wood material, in most cases chips, is introduced into a digester/digesters 2 and the chips are at least partly disintegrated by the cooking chemicals already in the digester 2 into fibers. This disintegration is based on the dissolution of the substances binding the fibers to each other, i.e. mainly lignin, into the

cooking solution. Today, a so-called digester wash is performed in most cases towards the end of the digestion process which aims at separating the cooking chemicals and the substances dissolved during the digestion process into the liquid phase, such as the lignin mentioned, from the pulp discharged from the digester 2. This kind of a wash is, however, not even close to complete but large volumes of cooking chemicals and substances mentioned above remain in the pulp. These are further removed mainly in the brown stock washing 8. The end result is that the dry-solids content of the pulp decreases relatively evenly from the digester 2 to the oxygen stage 10.

- 10 A problem, which has given rise to the invention, i. e. deterioration of the pulp quality in the oxygen stage, will be discussed in the following. Since the main purpose of the oxygen stage 10 is to decrease the Kappa number of the pulp, in other words mainly to dissolve the lignin still remaining in the fibers into the liquid phase, the dry-solids content of the liquid phase increases essentially in the oxygen stage 10. This dry-solids content of the liquid phase is decreased in the following wash 12 so that there would not be much extra impurities in the pulp in the bleaching stage following the oxygen stage. All the impurities ending up in the bleaching stage consume bleaching chemicals; thus it is profitable also in view of the chemical economy to separate these substances efficiently before the bleaching. For example, if the oxygen stage is followed in the process by an ozone stage, ozone will react with all dry substance it meets, i.e. also with any organic substance in the liquid phase. Thus all the ozone, which has reacted with any other material than with the lignin in the fibers has been consumed in inappropriate reactions and thus has not been made use of. Naturally, the same phenomenon applies also to other chemicals. Figure 7 illustrates the influence of COD on the consumption of the treatment chemical at different Kappa numbers of the pulp. The figure clearly indicates an increase in the consumption of the chemical when the COD increases, irrespective of the Kappa number. For this reason the washing stages between the bleaching stages, particularly the wash following the first oxygen stage, or in a broader sense the wash subsequent to the delignification or pre-bleaching stage, are arranged very efficient so as to minimize the consumption of bleaching chemicals in unnecessary reactions. Correspondingly, all bleaching stages are followed by one or more washer/s which aim at washing the reaction

products of the bleaching stage from the pulp as completely as possible before introduction of the subsequent bleaching chemical to the pulp.

However, primarily environmental factors, although in some cases also the volume of water available, have effectively limited the volume of water used in the washing stages. The ultimate goal is to provide a pulp mill with a closed water circulation. This means a situation where the mill uses the water it takes from a water system so efficiently that practically no water at all is discharged back to the water system but the liquid is circulated continuously inside the mill and fresh water is introduced only to replace the liquid evaporated in the process.

In order to accomplish the closed circulation, mills have almost without exception adopted the so-called counter-current wash method. This means that clean wash liquid, i.e. either water circulated inside the mill or water taken from a water system or these both are guided to be used as the wash liquid in the washing stage following the last bleaching stage of a fiber line. In other words "clean" wash liquid is transported to a point in which the pulp contains the smallest volume of dry solids or chemicals to be washed and in which the purity requirement is the greatest. From this point on the wash liquid is transferred counter-currently from one washer to another towards the digester/digestion plant so that while in each washing stage the dry-solids content of the pulp decreases the dry-solids content of the wash liquid recycled counter-currently increases.

The volume of impurities in the wash liquid has been found particularly problematic in connection with the oxygen stage. The reason for this is that a modern oxygen stage, particularly the two-stepped oxygen stage which is more and more often used, dissolves dry solids from fibers so efficiently that large volumes of dry solids end up in the filtrate in the washer following the oxygen stage. When this filtrate is transported to the washer preceding the oxygen stage to be used as wash liquid, most of the dry solids dissolved in the oxygen stage is returned to the pulp, and thus in the oxygen stage there are dry solids present both in the fibers and in the liquid phase surrounding the fibers. The volume of impurities increases in the circulation cumulatively until the volume reaches a balance,

which depends mainly on the efficiency of the washers, the dilution factor and the amount of impurity dissolved. This has been found to have a detrimental influence on the quality of the pulp. Primarily this means a distinct decrease of the pulp strength in the oxygen stage. This property is illustrated in Fig. 8, which depicts the influence of the COD content of the pulp on the decrease of viscosity of the pulp in the oxygen stage.

It is known from the prior art that a COD (Chemical Oxygen Demand) content in the liquid phase decreases the selectivity of delignification and bleaching and, as a consequence, the delignifying and bleaching chemicals react both with the lignin and the cellulose which results in a decrease in the pulp strength. It is further known from the prior art that the COD in the liquid phase varies when determined at different stages of the delignification. On one hand, it has been alleged that the type of the COD has no significance in view of the pulp strength but on the other hand, also the opposite has been argued. In other words, it has been alleged that a COD which has passed through a delignification stage has changed so that it has a stronger decreasing influence on the selectivity whereby recycling this kind of COD back to the delignification stage by counter-current washing would be wrong.

However, irrespective of the reason for the decrease in the pulp strength, i.e. a high COD content in the circulation waters, a wrong type of the COD in the oxygen stage or in general any physical property of the liquid phase, i.e. the amount of dry solids, the COD or the alkalinity, the present invention efficiently removes these problems.

Figure 2 illustrates a preferred embodiment of the invention for solving for example the problem discussed above. As it has been suspected that the reason for the problem is the high dry solids or COD content of the filtrate circulated from the washer 12 following the oxygen stage 10, a separator 114 has been provided in the line transporting filtrate from the washer 12 following the oxygen stage 10 to the washer 8 preceding the oxygen stage 10, the separator separating a partial liquid flow LI from the filtrate/wash liquid flow between the washers 8 and 12 for further treatment. The task of the separator 114 is to divide the filtrate flow LI to be treated into a cleaner fraction CC, i.e. a fraction having a lower dry solids or COD content, and into a fouler fraction CD, i.e. a fraction having a

higher dry solids or COD content, and to return the cleaner fraction CC to the wash liquid introduced into the washer 8 preceding the oxygen stage 10 as illustrated in Fig. 2. Then the dry solids or COD content of the filtrate in question, i.e. the wash liquid of the washer 8 preceding the oxygen stage 10, may be reduced remarkably so that the amount of dry solids or COD introduced into the oxygen stage 10 is essentially lower than before. Performed tests have confirmed that the quality of the pulp improves when the impurity or COD content decreases. The fouler fraction CD, or the fraction having a higher content of dry solids or COD, from the separator 114 is transported counter-currently so far that the dry solids or COD content of the liquid phase at that point is the same or higher than that of the fouler fraction CD to be returned. Locations of this kind may be for example the filtrate BSF flowing from the brown stock washing 8 to the digester wash of the digester 2, or black liquor, suitable internal liquid circulations of the digester 2, or black liquor flowing from the digester 2 to the chemical recovery CR.

It has been found out during studies that the same or sometimes even better effect on the quality of the pulp is obtained when an appropriate filtrate or other liquid containing liquor is treated closer to the digester. In this case also alkaline in the liquid phase comes into question. Further, the circulation contains as impurity also for example soap. It is a known fact that local separation of soap may be intensified by increasing the dry solids content of the liquid to an adequate extent. Fig. 9 illustrates the influence of the COD on the solubility of soap. As increasing the COD decreases the solubility of soap, soap is separated more easily onto the surface of the concentrate from which it may be removed by known methods. The removal of soap from the process improves the operability and the controllability of the whole process. Thus the present invention also provides a solution for the various problems caused by soap in different stages of the process.

Figure 3 illustrates a second and a third embodiment of the invention. In the embodiments of the figure, a separation device, in this embodiment an evaporator 214 changing a physical property of the liquid phase, such as dry solids content, COD, sodium or alkali content, has been provided in connection with the brown stock washing 8. A part LI of the filtrate transported from the washer 12 following the oxygen bleaching 10 to the brown stock washer 8 for use as the wash liquid is separated to the evaporator 214. In the

embodiment of the figure, the washer 12 is a so-called fractionating washer, which separates two filtrate fractions FC and FD, either of which is treated in the evaporator 214. Performed tests and calculations have proved that a maximum of 6 m³/adt of clean fraction may be used economically, usually about 1 – 5 m³, preferably 1 – 3.5 m³. Then the exact volume of the liquid flow directed into the evaporator from each of the flows cannot be stated because the volume of the filtrate LI to be brought to the evaporator depends also on the dry solids, COD and alkali content of the filtrate LI in question. The filtrate part LI is treated in the evaporator 214 so that condensate CC and concentrate CD are obtained. The basic principle is, as already explained earlier, that the concentrate CD is brought counter-currently to a point of the process where the foulness of the liquid phase, the volume of impurities, the dry solids, COD, or alkali content are the same as or greater than that of the concentrate CD. Correspondingly, the condensate CC is either returned to the point of the process from which the flow to the evaporator was extracted, or to some other later point (in the flow direction of the fiber suspension) in the process to be used as wash liquid.

In the embodiment of Fig. 3, the condensate CC from the evaporator 214 is returned to the same point from which it was extracted, i.e. to the feed of the brown stock washer 8. As the washer in the embodiment of the figure is a so-called DRUMDISPLACER[®] washer which uses wash liquids of various different degrees of purity, the condensate CC is returned to the feed line of the cleaner wash liquid FC. The concentrate CD, on the other hand, is guided to the black liquor flowing from the digester to the chemical recovery CR.

The figure illustrates with a broken line as a third preferred embodiment of the invention also another point where the condensate CC from the evaporator 214 may be returned. This point is the wash liquid feed of the washer 12 subsequent to the oxygen stage 10. Further, since the washer is a DRUMDISPLACER[®] washer, as the figure illustrates, the condensate CC may be returned to the cleaner wash liquid to be supplied to the washer 12.

Figure 4 illustrates preferred embodiments of the invention as applied to the digester of a continuous digestion process. The common feature with the embodiments of figure 4 is that the evaporator 314 treats either black liquor BSF transferred from the brown stock washing 8 to the digester 2 or black liquor transferred from the digester to the recovery CR.

In other word there are two alternative liquors introduced to the evaporator 314. The first one is to take a part LI from the filtrate transported from the brown stock washing 8 to the digestion plant to the so-called digester wash and to treat it in the evaporator 314. When a fractionating DRUMDISPLACER[®] washer is used as the brown stock washer, the fouler wash filtrate is brought in the embodiment of the figure from the washer 8 to the digester and the cleaner wash filtrate is directed to the bottom dilution of the blow tank 4. The filtrate BSF is used in the digester 2 in the so-called digester wash or in some other liquid circulation of the digester. The other alternative is to take to the evaporator 314 the part LI from the black liquor transported from the digester to the recovery CR. As it is quite feasible to use for example the apparatus of the embodiment illustrated in figure 3 combined with the apparatus of this embodiment, at least a part of the concentrate to be returned from the evaporator 214 of figure 3 may end up in the evaporator 314.

As regards alternative ways of returning the condensate from the evaporator 314 to the process, figure 4 illustrates six alternatives. According to one embodiment the condensate is returned to the cleanest wash liquid of the washer 12 subsequent to the oxygen bleaching 10, of course only if the condensate is cleaner than the fouled wash liquid. The second alternative is to return the condensate to the cleanest wash liquid of the brown stock washer 8 or to the final dilution if a press is used there. The third alternative is to return the condensate to the digester 2 for digester wash with the cleanest filtrate from the brown stock washer 8. The fourth alternative is to return the condensate to the bottom dilution of the blow tank 4. The fifth alternative is to return the condensate to the point in the bleaching plant BL where cleanliness is needed most. The wash filtrates are recycled in counter-current wash from the bleaching plant BL to the chemical recovery. The sixth alternative is to divide the condensate flow into two or more separate flows and to guide them to the locations described above.

Figure 5 illustrates in fact four further preferred embodiments of the invention as applied to the batch digestion process. In the embodiment of figure 5, the liquid LI supplied to the evaporation plant is obtained from either of the filtrate/wash liquid FC flowing from the washer (DRUMDISPLACER®) 12 following from the oxygen stage 10 to the brow stock washer 8. The feature determining the choice of the filtrate as well in this embodiment as in the others is the COD and dry solids content of the filtrate at that point. In most cases the filtrate having a higher content of COD and/or dry solids is chosen. Further, as illustrated in figure 5, the condensate CC is returned the wash liquid flowing to the brown stock washer 8. Preferably to the same wash liquid FC from which the liquid LI to be supplied to the evaporator 414 is obtained. Another alternative of returning the condensate CC is to direct it to a location far in the process, even after the oxygen stage in the flow direction of the fiber suspension. Figure 5 illustrates a process where the screening plant 6 has been positioned after the oxygen stage 10. In this case it has been considered advantageous to direct the condensate CC to the wash liquid of the washing stage 16 subsequent to the screening plant 6. Again it may be stated that, if wash liquids of different concentrations are supplied to the washer, it is advantageous to return the condensate CC to the cleaner wash liquid, i.e. in practice to the last wash stage or zone of the washer. It would even be advantageous to transport the condensate CC as a separate flow to the end of the washing stage or to the end dilution of a press.

In the embodiment of figure 5, the concentrate CD is returned either to the black liquor flowing from the digester/digestion plant 2 to the chemical recovery CR, or to the filtrate BSF flowing from the brown stock washing 8 to the digestion wash of the digester 2 or to some other liquid circulation of the digester.

Figure 6 illustrates six further preferred embodiments of the invention as applied to the batch digestion process. In the embodiment of the figure, the black liquor LI supplied to the evaporator 514 is obtained either from the flow BSF flowing from the brow stock washing 8 to the digestion plant, from the flow from the brown stock washing 8 to the chemical recovery CR, or from the flow from the digestion plant 2 to the chemical recovery CR. The condensate CC in turn is added either to the wash liquid coming to the

brown stock washer 8, in case of a DRUMDISPLACER[®] washer to the cleaner wash liquid FC coming to the washer 8, or in this embodiment even to the wash liquid of the washer subsequent to the oxygen stage 10 and the screening 6. The concentrate CD from the evaporator 514 in turn is returned directly to the flow to the chemical recovery CR.

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Yet another application of or an alternative way of using the method of the invention is the separation treatment of the white liquor coming from the chemical recovery to the digester. Quite like in the examples described above, the white liquor may be separated for example by evaporating into condensate and concentrate and the condensate may be brought to the same points of the process as in the previous embodiments. The concentrate in turn is supplied to the digester as more concentrated white liquor, i.e. to the same place where it would be supplied anyway.

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All the embodiments described above employ without exception a DRUMDISPLACER[®] washer as the washer which has the typical feature that wash liquids of several different concentrations may be supplied to it and filtrates of several different concentrations may be obtained from it. Further, it is characteristic of the washer in question that it may comprise several wash stages whereby the liquid circulations between the wash stages have been arranged by connections within the washer as described in various patents and patent applications discussing the subject. The corresponding function may at least partly be effected for example by means of suction drum filters or presses which in practice means that several suction drum filters or presses are connected one after the other. In this case it is possible to extract liquid for the evaporation treatment also from the filtrate/wash liquid lines between the filters/presses connected in series. In other words, the DRUMDISPLACER[®] washer is not indispensable in carrying out the invention but the invention may be used in connection with all washing apparatus available on the market. Thus it is clear that the invention is applicable also in situations where only one kind of wash liquid can be supplied to the washer and only one kind of filtrate can be extracted from the washer.

It should be noted that although the invention has been described in connection with an oxygen stage used as a delignification or prebleaching stage, the invention may be used

in connection with any kind of treatment stages. Thus the invention may quite well be employed in connection with a delignification stage using peroxide and oxygen together or chlorine dioxide although the use of chlorine dioxide sets certain limitations to recycling of the concentrate.

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Further, it is worth mentioning that although the above description speaks about an evaporator as the separating device in practice there are also other possibilities.

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For example in cases where absolute purity of the fraction CC to be recycled is not important, a membrane separator may be used which separates macromolecular dry solids and/or COD from the liquid to be recycled. Then dry solids and/or COD having smaller molecules will remain in the cleaner fraction CC but when this fraction CC is recycled to a suitable location in the process it does not cause essential problems.

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Further, it should be remembered that the cleaner fraction obtained from the separation apparatus may be distributed not only to one location as described above but also to several locations. An example of the many alternatives is to take a portion of the cleaner fraction CC to the washer of the oxygen stage and another portion to the washer preceding a PO stage (PO = peroxide bleaching stage intensified with oxygen) of the bleaching plant. Then, in the end the cleaner fraction brought to the process will end up counter-currently to the digester. The test we have performed have shown that the cleaner fraction could be returned to a point from which it would have to travel counter-currently through as many washes/washing stages as possible.

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We claim:

1. A method of treating pulp according to which method wood material is supplied into a digester (2), the so-called brown stock is discharged from the digester to brown stock washing (8) and the washed pulp is treated in a delignification stage (10) whereby the pulp digestion and brown stock washing processes mainly employ counter-current washing in which clean wash liquid is brought to the end of the process and transferred counter-currently relative to the flow direction of the fiber suspension through several washing stages at least partly to the digester (2) and from there further to chemical recovery CR, characterized in that in order to lower the COD-level in the oxygen delignification stage (10),
- a) a portion LI is separated before the process stage subsequent to the delignification stage (10) and the washer (12) of the delignification stage from the wash liquid/filtrate to be recycled counter-currently;
- b) the portion LI of the filtrate is treated in a separation device (114, 214, 314, 414, 514) in order to produce two fractions CC and CD having different physical properties;
- c) the fraction CC having a lower physical property is returned either substantially to the same point in the process from which the portion LI of the filtrate was extracted at stage (a), or to some other process stage in order to lower the COD-level in the oxygen delignification stage;
- d) the fraction CD having a higher physical property is directed either to the flow passing to the chemical recovery CR, the digestion plant or as such to a point in the process in which the dry-solids, COD and /or alkali content of the liquid phase is at least as high as that of the fraction CD.
2. A method as claimed in claim 1, characterized in that the filtrate LI of stage a) is obtained from the flow passing from the digestion plant (2) to the chemical recovery CR and the fraction CD of stage d) is returned to the flow passing to the chemical recovery CR.

3. A method as claimed in claim 2, characterized in that the fraction CC of stage c) is returned either to the flow BSF passing from the brown stock washing (8) to the digester (2), or to be used as the wash liquid in the brown stock washer (8), in the washer (12) following the delignification stage (10), or in the washer (16) following the screen plant (6).

4. A method as claimed in claim 1, characterized in that the filtrate LI of stage a) is obtained from the filtrate flow passing to the brown stock washer (8) preceding the delignification stage (10).

5. A method as claimed in claim 4, characterized in that the fraction of stage c) is returned to the wash liquid flow passing to the brown stock washer (8) and the fraction of stage d) is returned either to the flow BSF passing from the brown stock washer (8) to the digester (2), or directly to the flow passing to the chemical recovery CR.

6. A method as claimed in claim 1, characterized in that at stage a) the filtrate LI to the separation device is taken from the circulation waters subsequent to the digester (2), the fraction CD of stage d) is passed to the liquid circulations of the digester (2) or directly to the chemical recovery CR, and the fraction CC of stage c) is returned to be used as the wash liquid in the brown stock washing (8) or in the wash (12) subsequent to the delignification stage (10).

7. A method as claimed in any of the preceding claims, characterized in that at least one of the washers (8, 12, 16) is a washer or a press from which at least either at least two filtrates (FC, FD) having different physical properties are extracted or to which at least two filtrates having different physical properties are introduced.

8. A method as claimed in claim 7, characterized in that the fraction of stage c) is returned to be used as the wash liquid in the washer or press in question with the wash liquid FC introduced thereto and having the lower physical property.

9. A method as claimed in claim 7, characterized in that the filtrate LI of stage a) is taken from at least one filtrate FC of the washer or press in question.
10. A method as claimed in claim 7, characterized in that the filtrate LI of stage a) is taken from at least one filtrate FC of the washer or press in question having the higher physical property.
11. A method as claimed in claim 1, characterized in that the separation device (114, 214, 314, 414, 514) is a membrane separator.
12. A method as claimed in claim 1, characterized in that the separation device is an evaporator (114, 214, 314, 414, 514), whereby the fraction having the lower physical property is condensate and the fraction having the higher physical property is concentrate.
13. A method as claimed in claim 1, characterized in that the volume of the fraction CC having the lower physical property returned at stage c) from the separation treatment is $6 \text{ m}^3/\text{adt}$ at the most, about $1 - 5 \text{ m}^3/\text{adt}$, preferably $1 - 3.5 \text{ m}^3/\text{adt}$.
14. A method as claimed in claim 1, characterized in that the liquid to be treated in stage b) is white liquor flowing from the chemical recovery to the digestion plant (2).
15. A method as claimed in claim 1, characterized in that soap is separated from the fraction obtained from stage b) and having the higher dry solids content.
16. A method as claimed in claim 1, characterized in that pulp is further treated in the bleaching stages BL following the delignification so that at least part of the fraction CC to be returned at stage c) is passed to a washer or press of a bleaching stage.
17. A method as claimed in claim 16, characterized in that also at least a part of the wash liquids used in the bleaching BL is passed counter-currently up to the digestion plan (2).

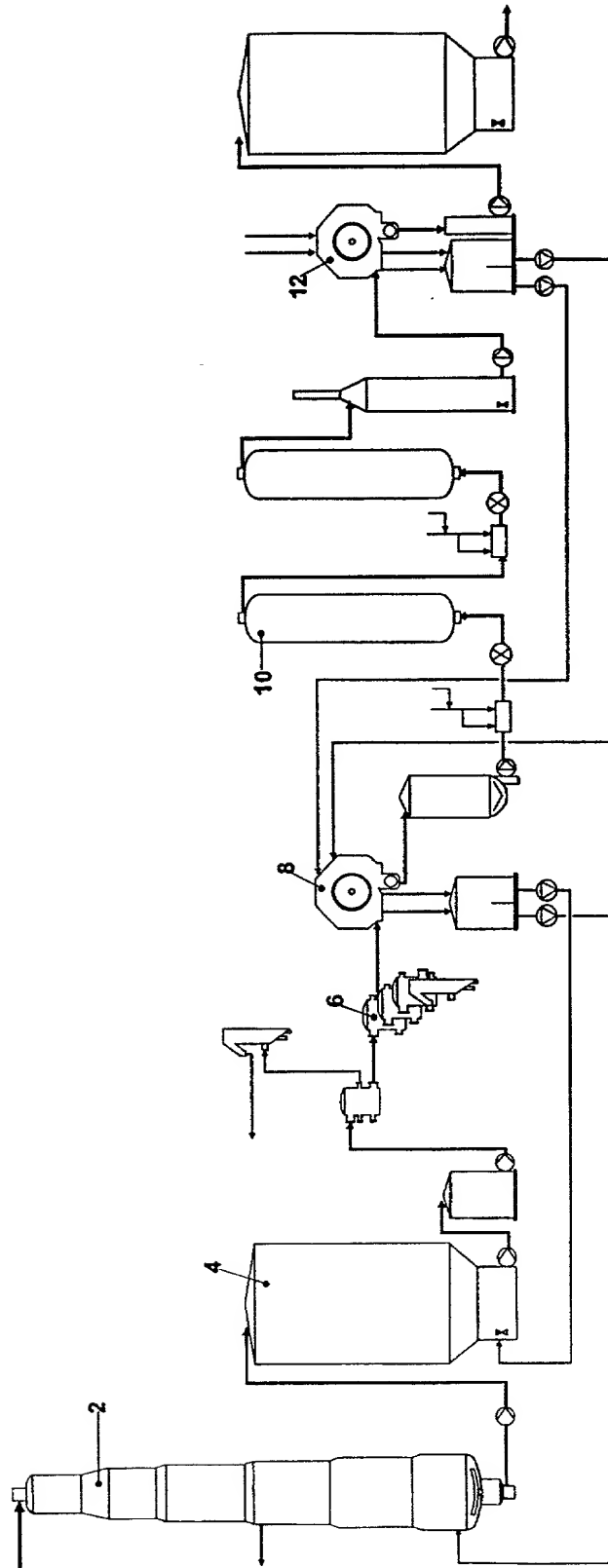


Fig. 1

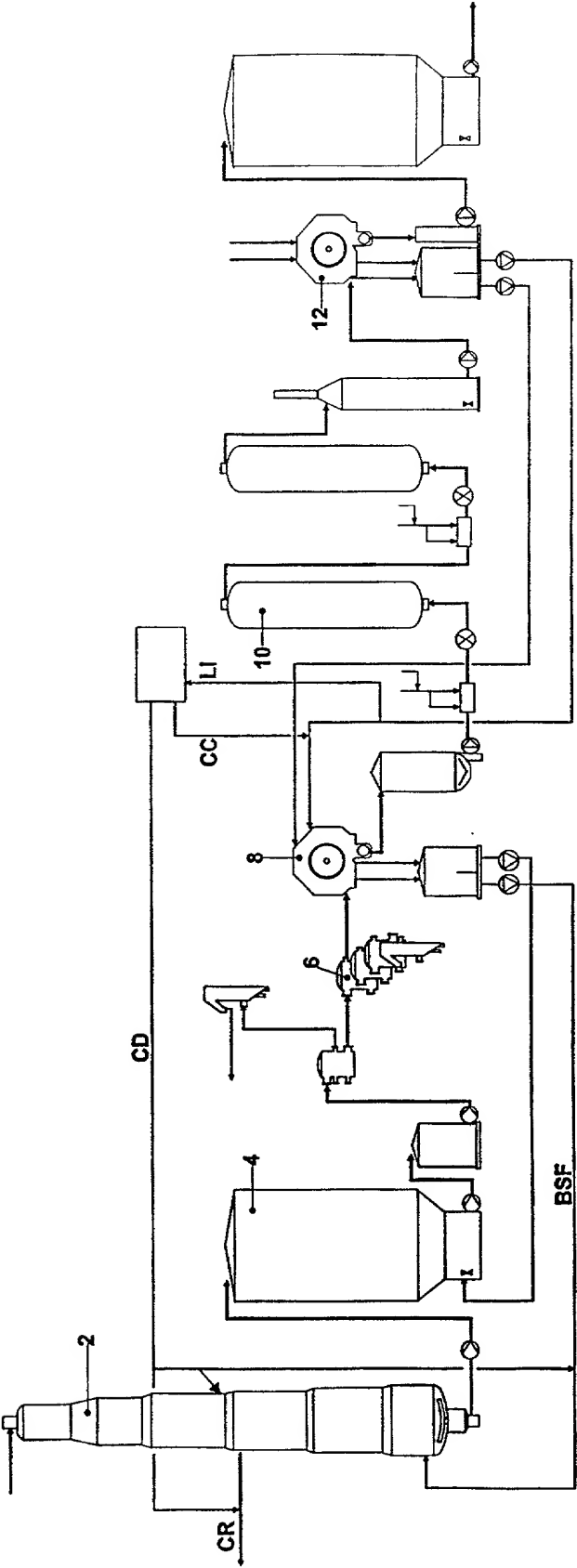


Fig. 2

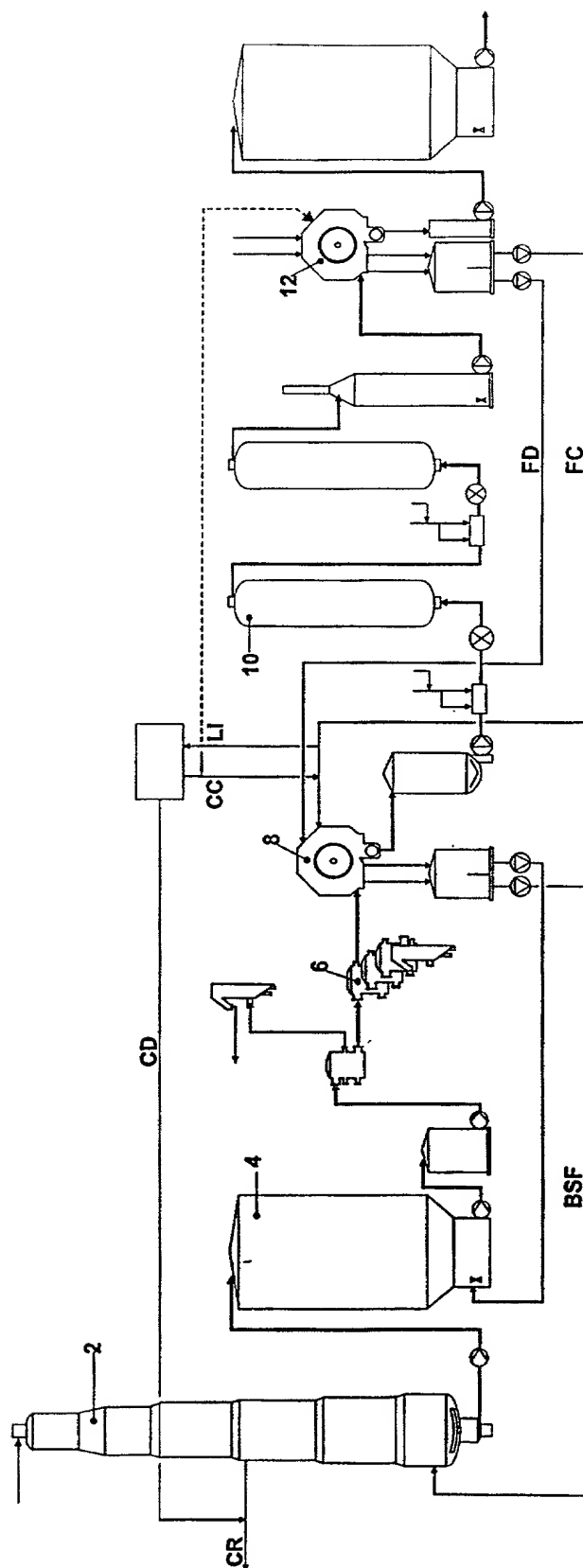


Fig. 3

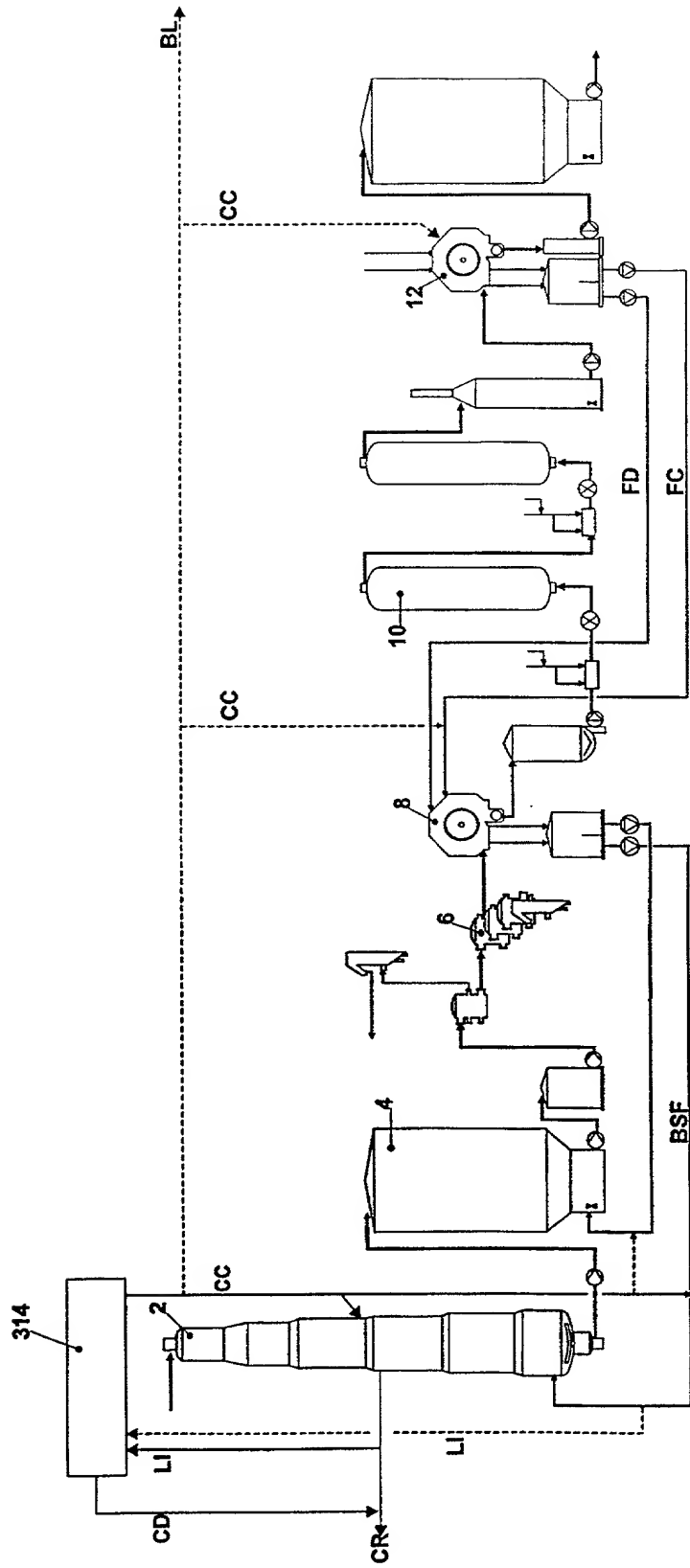


Fig. 4

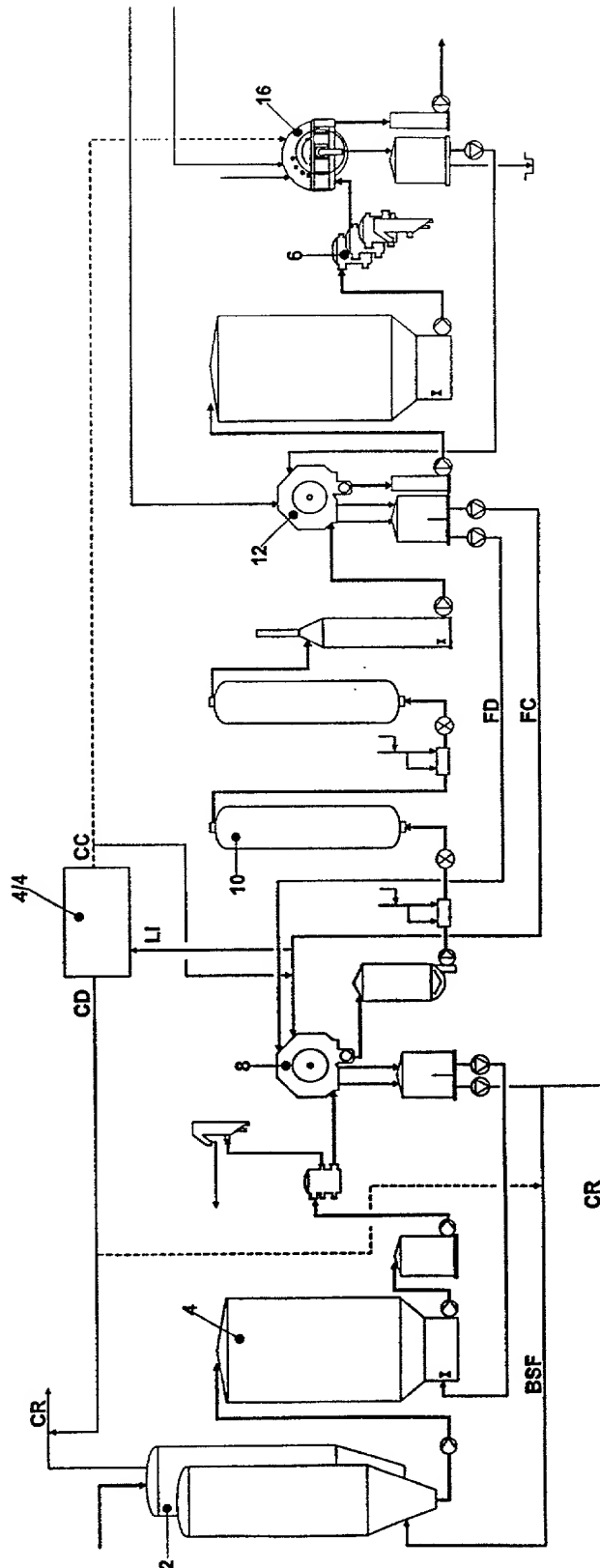


Fig. 5

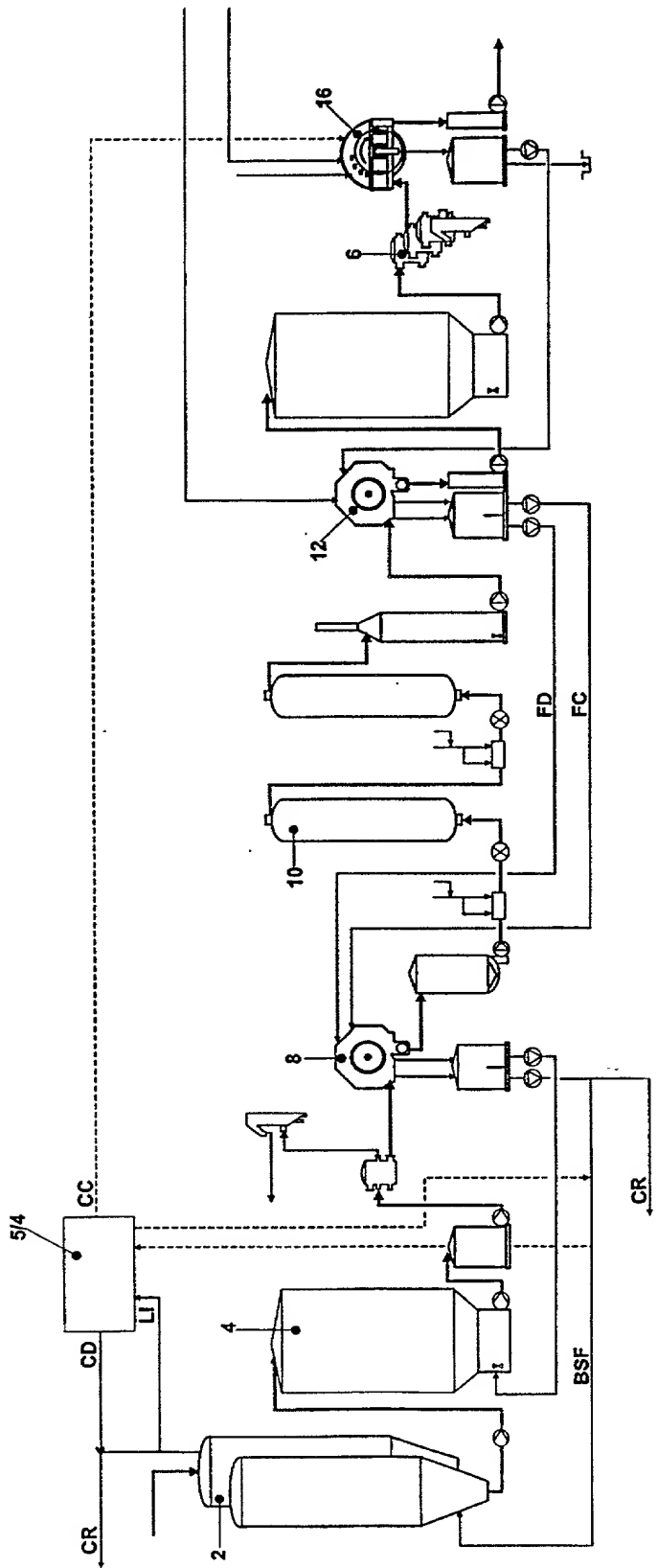


Fig. 6

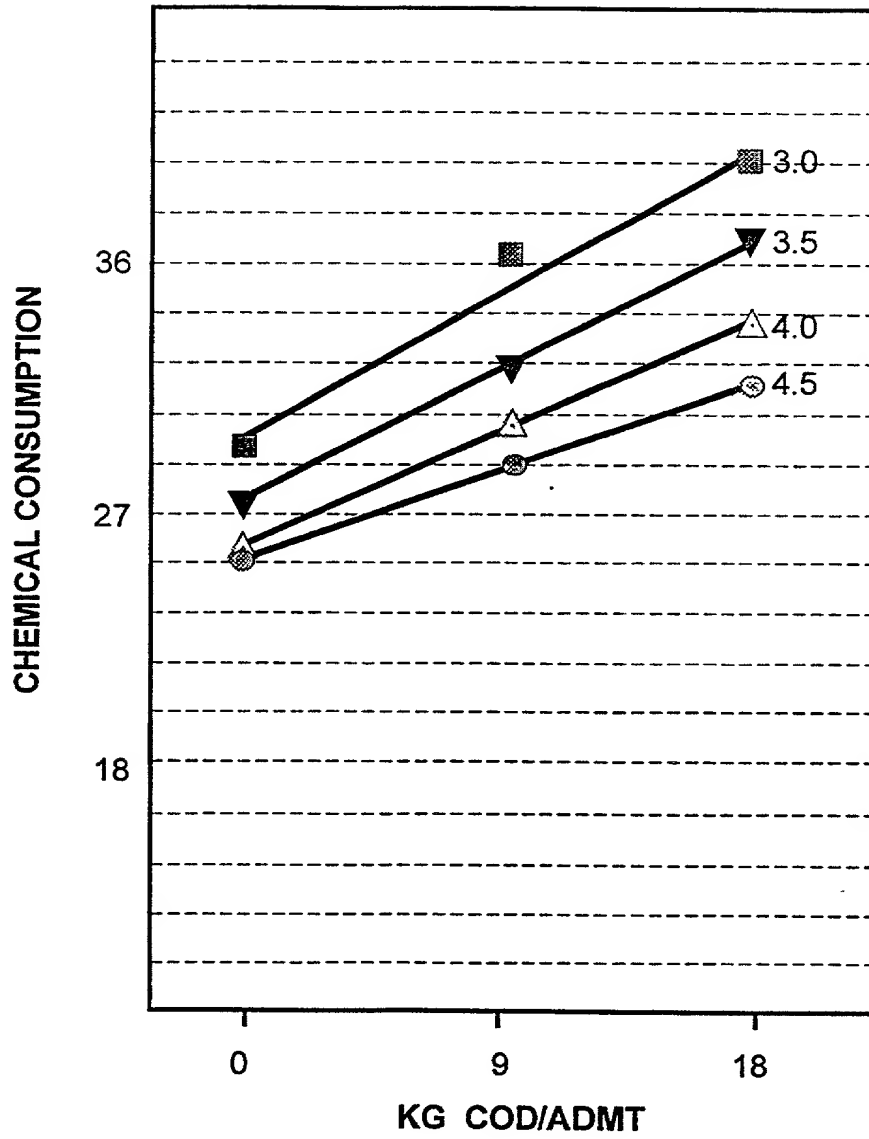


Fig. 7

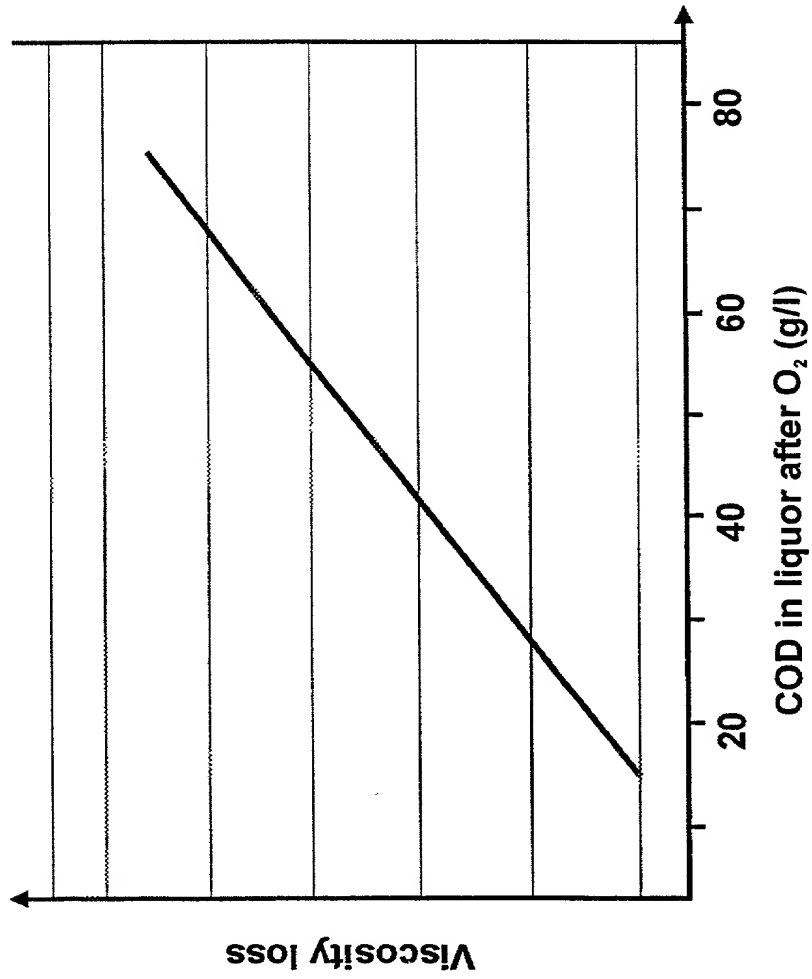


Fig. 8

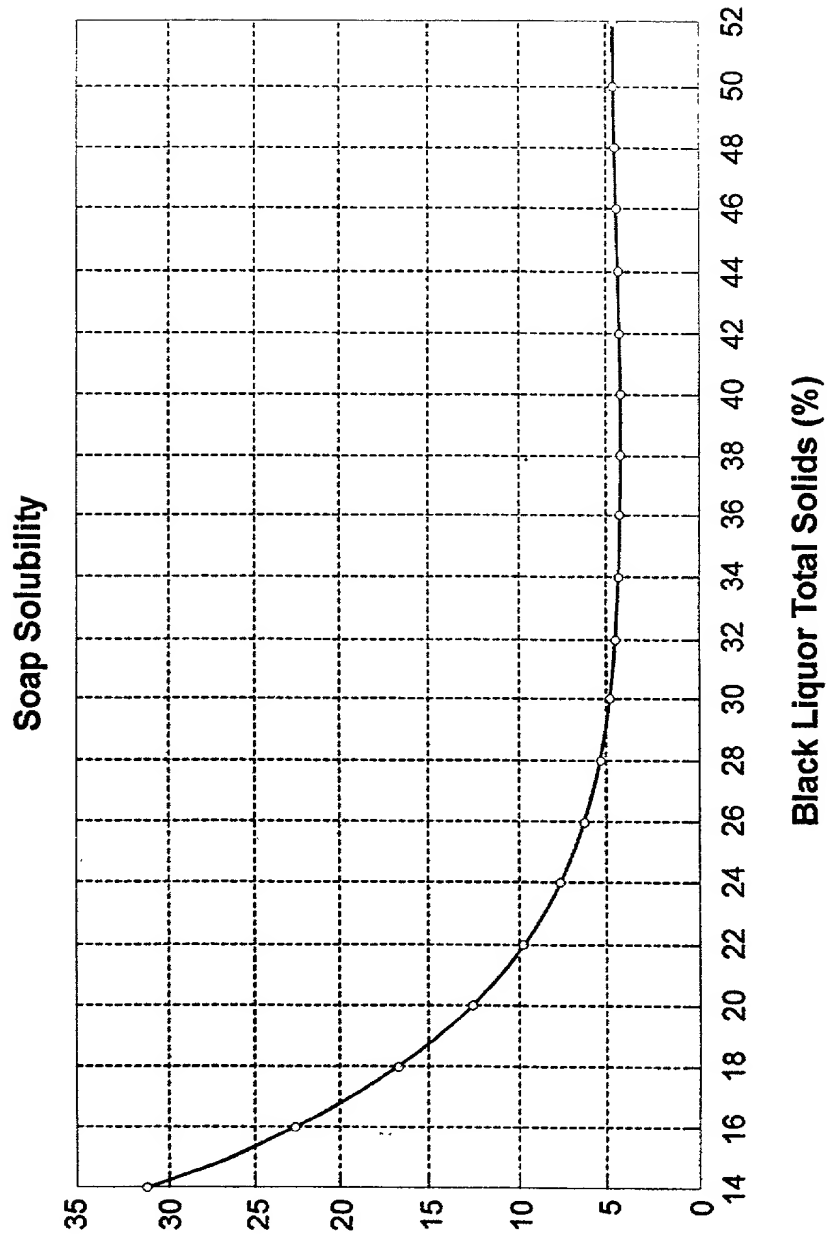


Fig. 9

#3

RULE 63 (37 C.F.R. 1.63)
INVENTORS DECLARATION FOR PATENT APPLICATION
IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

As a below named inventor, I hereby declare that my residence, mailing address and citizenship are as stated below next to my name, and I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled:

TREATMENT OF RECIRCULATED FILTRATES IN PULP PRODUCTION

the specification of which (check applicable box(es)):

☐ is attached hereto
☐ was filed on _____ as U.S. Application Serial No. _____ (Atty Dkt. No. 30-551)
☒ was filed as PCT International application No. PCT/FI00/00045 on 20 January 2000
and (if applicable to U.S. or PCT application) was amended on June 20, 2000 And February 15, 2001

I hereby state that I have reviewed and understand the contents of the above identified specification, including the claims, as amended by any amendment referred to above. I acknowledge the duty to disclose to the Patent Office all information known to me to be material to patentability as defined in 37 C.F.R. 1.56. I hereby claim foreign priority benefits under 35 U.S.C. 119/365 of any foreign application(s) for patent or inventor's certificate listed below and have also identified below any foreign application for patent or inventor's certificate having a filing date before that of the application on which priority is claimed or, if no priority is claimed, before the filing date of this application:

Application Number	Country	Day/Month/Year Filed
990127	Finland	22 January 1999

I hereby claim the benefit under 35 U.S.C. §119(e) of any United States provisional application(s) listed below.

Application Number	Date/Month/Year Filed
--------------------	-----------------------

I hereby claim the benefit under 35 U.S.C. 120/365 of all prior United States and PCT international applications listed above or below:

Prior U.S./PCT Application(s):	Day/Month/Year Filed	Status: patented pending, abandoned Pending
Application Serial No. PCT/FI00/00045	20 January 2000	

29 I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon. And on behalf of the owner(s) hereof, I hereby appoint **NIXON & VANDERHYE P.C., 1100 North Glebe Rd., 8th Floor, Arlington, VA 22201-4714**, telephone number (703) 816-4000 (to whom all communications are to be directed), and the following attorneys thereof (of the same address) individually and collectively owner's/owners' attorneys to prosecute this application and to transact all business in the Patent and Trademark Office connected therewith and with the resulting patent: Larry S. Nixon, 25640; Arthur R. Crawford, 25327; James T. Hosmer, 30184; Robert W. Faris, 31352; Richard G. Besha, 22770; Mark E. Nusbaum, 32348; Michael J. Keenan, 32106; Bryan H. Davidson, 30251; Stanley C. Spooner, 27393; Leonard C. Mitchard, 29009; Duane M. Byers, 33363; Jeffry H. Nelson, 30481; John R. Lastova, 33149; H. Warren Burnam, Jr. 29366; Mary J. Wilson, 32955; J. Scott Davidson, 33489; Alan M. Kagen, 36178; Robert A. Molan, 29834; B. J. Sadoff, 36663; James D. Berquist, 34776; Updeep S. Gill, 37334; Michael J. Shea, 34725; Donald L. Jackson, 41090; Michelle N. Lester, 32331; Frank P. Presta, 19828; Joseph S. Presta, 35329; Joseph A. Rhoads, 37515; Raymond Y. Mah, 41426; Chris Comuntzis, 31097. I also authorize Nixon & Vanderhye to delete any attorney names/numbers no longer with the firm and to act and rely solely on instructions directly communicated from the person, assignee, attorney, firm, or other organization sending instructions to Nixon & Vanderhye on behalf of the owner(s).

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RULE 63 (37 C.F.R. 1.63)
INVENTORS DECLARATION FOR PATENT APPLICATION
IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

As a below named inventor, I hereby declare that my residence, mailing address and citizenship are as stated below next to my name, and I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled:

TREATMENT OF RECIRCULATED FILTRATES IN PULP PRODUCTION

the specification of which (check applicable box(es)):

☐ is attached hereto
☐ was filed on _____ as U.S. Application Serial No. _____ (Atty Dkt. No. 30-551)
☒ was filed as PCT International application No. PCT/FI00/00045 on 20 January 2000
and (if applicable to U.S. or PCT application) was amended on June 20, 2000 And February 15, 2001

I hereby state that I have reviewed and understand the contents of the above identified specification, including the claims, as amended by any amendment referred to above. I acknowledge the duty to disclose to the Patent Office all information known to me to be material to patentability as defined in 37 C.F.R. 1.56. I hereby claim foreign priority benefits under 35 U.S.C. 119/365 of any foreign application(s) for patent or inventor's certificate listed below and have also identified below any foreign application for patent or inventor's certificate having a filing date before that of the application on which priority is claimed or, if no priority is claimed, before the filing date of this application:

Priority Foreign Application(s):

Application Number	Country	Day/Month/Year Filed
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I hereby claim the benefit under 35 U.S.C. §119(e) of any United States provisional application(s) listed below.

Application Number	Date/Month/Year Filed
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I hereby claim the benefit under 35 U.S.C. 120/365 of all prior United States and PCT international applications listed above or below:

Prior U.S./PCT Application(s):

Application Serial No.	Day/Month/Year Filed	Status: patented pending, abandoned Pending
PCT/FI00/00045	20 January 2000	

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon. And on behalf of the owner(s) hereof, I hereby appoint NIXON & VANDERHYE P.C., 1100 North Glebe Rd., 8th Floor, Arlington, VA 22201-4714, telephone number (703) 816-4000 (to whom all communications are to be directed), and the following attorneys thereof (of the same address) individually and collectively owner's/owners' attorneys to prosecute this application and to transact all business in the Patent and Trademark Office connected therewith and with the resulting patent: Larry S. Nixon, 25640, Arthur R. Crawford, 25327; James T. Hosmer, 30184; Robert W. Faris, 31352; Richard G. Besho, 22770; Mark E. Nusbaum, 32348; Michael J. Keenan, 32106; Bryan H. Davidson, 30251; Stanley C. Spooner, 27393; Leonard C. Mitchard, 29009; Duane M. Byers, 33363; Jeffrey H. Nelson, 30481; John R. Lastova, 33149; H. Warren Burnam, Jr. 29366; Mary J. Wilson, 32955; J. Scott Davidson, 33489; Alan M. Kagen, 36178; Robert A. Molan, 29834; B. J. Sadoff, 36663; James D. Berquist, 34776; Updeep S. Gill, 37334; Michael J. Shea, 34725; Donald L. Jackson, 41090; Michelle N. Lester, 32331; Frank P. Presta, 19828; Joseph S. Presta, 35329; Joseph A. Rhoads, 37515; Raymond Y. Mah, 41426; Chris Comuntzis, 31097. I also authorize Nixon & Vanderhye to delete any attorney names/numbers no longer with the firm and to act and rely solely on instructions directly communicated from the person, assignee, attorney, firm, or other organization sending instructions to Nixon & Vanderhye on behalf of the owner(s).

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